(19) Europäisch s Patentamt
European Patent Offic
Office européen d s brevets

(11) EP 0 744 634 B1

(12) EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent: **08.01.2003 Bulletin 2003/02**

(51) Int Cl.[7]: **G02B 5/30**, G02F 1/09, G02B 1/08

(21) Application number: **96303673.6**

(22) Date of filing: **23.05.1996**

(54) Method of producing an optical polarizer

Methode zu Herstellung eines optischen Polarisators

Méthode de fabrication d'un polarisateur optique

(84) Designated Contracting States:
DE FR SE

(30) Priority: **23.05.1995 JP 12393795**
26.12.1995 JP 33977095

(43) Date of publication of application:
27.11.1996 Bulletin 1996/48

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Field of the Invention

[0001] The present invention relates to a production method of a polarizer wherein anisotropic metal particles are dispersed in a dielectric.

Prior Art

[0002] Polarizers are used for obtaining polarized light in a specific direction, and are used in optical communication, photosensors, optical interferometers, etc. For example, in the case of optical communication. polarizers are principal components of the optical isolator. The optical isolator comprises a first polarizer, a Faraday rotator and a second polarizer arranged in a holder, with magnets being arranged coaxially around them. The holder is made of, for example, a Ni-Fe alloy, etc., and the polarizers are joined onto the holder with sealing glass or solder and sealed hermetically. With regard to the polarizing performance, the value relative to the wavelength used for optical communication is important. The optical isolator is used in combination with a laser diode, etc.; the first polarizer polarizes light in a specific direction and takes it out, the Faraday rotator rotates the direction of polarization, and the second polarizer further rotates the direction of polarization and takes out the polarized light.

[0003] Polarizers in current use are mainly those wherein spheroidal silver particulates are dispersed in glass (the Japanese Patent Publication Hei-2-40619 and corresponding U.S. Patents 4,486,213 and 4,479,819). This type of polarizer is made by heat-treating a glass substrate containing silver and halogen to precipitate particulates of silver halide, and drawing the glass substrate on heating to stretch the silver halide particulates into spheroids. This process makes the silver halide particulates. Next, the glass substrate is heated in a reducing environment to reduce silver halide to metal silver. In this polarizer, however, the aspect ratios are not homogeneous; it is hard to precipitate silver particulates of which major axes and minor axes are homogeneous. Moreover, it is difficult to reduce residual silver halide in the glass, hence opaque silver halide remains. Furthermore, because the glass shrinks in the course of the reduction of silver halide, the glass surface becomes porous, resulting in a drop in the long-term stability.

[0004] EP-A-0 719 742 discloses a polarising glass article comprising a glass layer which contains shape-anisotropic metallic particles dispersed therein in an oriented state, and a transparent base glass. The polarising layer of this invention is also formed using the above technique. US 4,282,022 discloses a process for producing polarising materials comprising extruding glasses containing metallic silver particles so that the particles become elongated and aligned. Again, the metallic silver particles are produced by reducing precipitated silver halide particles.

[0005] To solve such problems, it has been proposed to produce polarizers by applying thin film processes such as vacuum evaporation and sputtering (Denshi Joho Tsushin Gakkai, Autumn General Meeting of 1990. Preprint C-212). According to this proposal, a metal layer is deposited on a dielectric substrate such as glass by vacuum evaporation, and a dielectric layer such as glass is made on the metal layer by sputtering. etc. In this way. several metal layers and dielectric layers are formed alternately. Then the substrate is drawn on heating to deform the metal layers into layers of discontinuous and insular metal particulates. The metal particulates of the metal particulate layers are drawn in the drawing direction to become spheroidal and exhibit a polarizing capability.

[0006] The present inventors, however, identified the following problems in the polarizer using the thin film processes:

1) Since it is difficult to precipitate large metal particulates, the extinction ratio between the polarizing direction and a direction perpendicular to it is low; and

2) As the extinction ratio is low, it is necessary to increase the extinction ratio by increasing the number of layers, but the layers may peel off the substrate when the number of layers is increased.

Summary of the Invention

[0007] The task of the present invention is to provide polarizers which have high extinction ratios and are free of peeling from the substrate, and to provide optical isolators using these polarizers.

[0008] The present invention is a production method for an optical polarizer according to claim 1.

[0009] It is sufficient for the substrate to be transparent, and any dielectric may be used, but preferably a substrate and a dielectric virtually of the same kind are used to have one common coefficient of thermal expansion for both the substrate and the dielectric. This is effective in preventing the polarizing layer from peeling off from the substrate. For the substrate, glass is preferable, which is inexpensive, easily drawable and transparent. Borosilicate glass is preferable, which has a coefficient of thermal expansion similar to that of a Ni-Fe alloy. Hence both said dielectric substrate and said dielectric of said polarizing layer are made of borosilicate glass.

[0010] For the metal particulates, a metal which can easily coagulate, has poor wettability with glass and is oxidized with difficulty is preferable. At least one material is preferably selected from a group comprising, for example, precious metals, Cu, Fe, Ni and Cr. The most preferable materials are Au, which has a low melting point, has poor wettability with glass and is oxidized with difficulty, and Cu, which is inexpensive. The metal content in the polarizing layer is preferably from 5 to 15 %

by volume, and in this range, the metal particulates do not form a different phase, and the metal content in the polarizing layer can be raised to a relatively high level to increase the extinction ratio.

[0011] The aspect ratio of the metal particulates is preferably from 10 to 30 in average, and most preferably from 15 to 25. The mean major axis length of the metal particulates is preferably from 10 to 300 nm, and more preferably from 30 to 200 nm, and most preferably from 40 to 200 nm. The mean minor axis length of the metal particulates is preferably from 1 to 10 nm, and more preferably from 2 to 10 nm.

[0012] To produce such a polarizer, for example, a thin film of a mixture of a dielectric and a metal is formed on at least one main face of a transparent dielectric substrate, then said thin film mixture is heated to make the metal in the thin film mixture coagulate to form metal particulates, after that, said substrate and said thin film mixture are drawn on heating to stretch said metal particulates into spheroids.

[0013] Here, preferably, only a single layer of said mixture thin film is formed. When a thin film mixture of a dielectric and a metal is used, a single layer can provide a sufficient extinction ratio: use of multiple layers has a possibility of peeling off of the polarizing layer. Such a polarizer is combined with, for example, a Farady rotator, a magnet and a holder to make an optical isolator.

[0014] According to the present invention, a thin film mixture of a metal and a dielectric is deposited, and this thin film mixture is heated to coagulate the metal. This heating temperature is preferably lower than the softening point of the dielectric substrate. When the thin film mixture is heated, the metal particulates will coagulate with each other to form relatively large metal particulates. Next, when the substrate and the thin film mixture are drawn, the metal particulates will be stretched in the drawing direction to acquire anisotropy. As a result, the metal particulates become spheroidal. Let us compare the polarizer according to the present invention with the prior art wherein metal thin films and dielectric thin films are deposited alternately. In the prior art, because a dielectric thin film is present between two metal thin films, metals do not coagulate across the dielectric thin films; thus large metal particulates hardly grow. As a result, the extinction per one layer is small, and many layers must be deposited. The present inventors have found that the greater the number of the deposited layers, and the greater the total film thickness of the polarizing layer, the greater is the possibility for the polarizing layer of peeling off from the substrate. In contrast, according to the present invention, because a mixture thin film is used, it is sufficient to deposit a single layer, and no peeling will happen. The present inventors have also found that according to the present invention peeling does not occur even when the film thickness of the polarizing layer is increased to 1μm or over.

Brief Description of the Drawings

[0015] Fig. 1 is a perspective view showing the polarizer produced according to an embodiment.

[0016] Fig. 2 is a characteristic diagram showing the extinction characteristic before drawing of the polarizer produced according to the embodiment.

[0017] Fig. 3 is a characteristic diagram showing the extinction characteristic after drawing of the polarizer produced according to the embodiment.

Embodiment

[0018] Fig. 1 through Fig. 3 show a polarizer 1 produced according to the embodiment. In Fig. 1, 2 denotes a glass substrate. For example, borosilicate glass such as Pyrex glass (Pyrex is a trade mark of Corning Glass Industry, Inc.) and BK glass (BK is a trade name of HOYA Corporation) may be used. In addition to them, high melting point glasses such as silica glass and low melting point glasses such as soda glass may be used. In place of the glass substrate 2, another transparent material may be used, but glass is inexpensive and can be easily drawn. In the present specification, transparency means that the material is transparent to the wavelength used. Of various glass materials, borosilicate glass is suited to the substrate 2, since the coefficient of cubical expansion of borosilicate glass is close to the coefficient of cubical expansion ($90\sim96\times10^{-7}$/ °C) of the Ni-Fe alloy to be used for the holder of the optical isolator and borosilicate glass can easily seal the holder. For example, the coefficient of cubical expansion of BK7 glass is $72\sim89\times10^{-7}$/°C.

[0019] 3 denotes a polarizing layer wherein spheroidal metal particulates 5 are almost homogeneously dispersed in a dielectric 4. The material of the dielectric 4 is desirably the same material as that of the substrate 2; for example, if Pyrex is used for the substrate 2, then Pyrex is desirably used for the dielectric 4 to match their coefficients of cubical expansion. The metal particulates 5 are preferably precious metals such as Au, Ag, Pt, Rh and Ir or transition metals such as Cu, Fe, Ni and Cr. Preferable metals are those that have bad wettability with the dielectric 4 and are easy to coagulate, and more over, that are hardly oxidized and can be present as metal particulates 5 in the dielectric 4. Of these materials, specially preferable ones are Au, which is easy to coagulate because of its low melting point, has a bad wettability with glass and is hardly oxidized, and Cu, which is inexpensive and has a bad wettability with glass.

[0020] The content of the metal particulates in the polarizing layer 3 is preferably from 5 to 15 % by volume. The higher is the content, the greater is the extinction ratio, but if the content exceeds 15 %, separation of the phase of the dielectric and that of the metal (metal particulate diameter is about 0.6μm) will occur and the polarizer can not be produced.

[0021] With regard to the thickness of the polarizing

layer, there are no special restrictions; for example, in the embodiment, the film thickness before the drawing was 0.5 μm. In contrast to it, in the prior art wherein multiple metal layers and dielectric layers are deposited, there is a limit to the total thickness of these layers. When the total thickness before the drawing exceeded 0.5 μm, peeling off from the substrate 2 took place. In the prior art, to improve the polarizing performance, it is necessary to increase the number of layers, and the resulting increase in the total film thickness will result in the peeling. The peeling of the polarizing layer, if it does occur, will take place during the heat treatment before the drawing; a bubble-like swell will occur over a wide area in the interface between the substrate and the metal layer or in the interface between a metal layer and a dielectric. According to the experiments made by the present inventor, it was found that the peeling of the polarizing layer is related to the formation thereof by depositing a large number of layers and the peeling does not occur if the polarizing layer is formed of a single layer just as the embodiment in which a mixture film of a dielectric and a metal is used.

[0022] The metal particulates 5 are spheroidal and have an anisotropy. For example, as shown in Fig. 1, the direction the light advances is defined as the direction Z, and a plane perpendicular to that is defined as the X-Y plane. In Fig. 1, the direction of the major axis of the metal particulates 5 is the direction Y,and the direction of the minor axis is the direction X. The ratio of the major axis length to the minor axis length of the metal particulate 5 is defined as the aspect ratio, and in this specification the average of the aspect ratios of many metal particulates 5 is simply called the aspect ratio. The metal particulates 5 become spheroidal because the metal particulates 5 are stretched together with the substrate 2 in the drawing direction during the drawing after the formation of the film of the polarizing layer 3. The higher is the aspect ratio, the greater is the extinction ratio; at the same time, however, the drawing rate of the substrate 2 also increases, making it difficult to draw, and moreover, the rate of increase in the extinction ratio decreases in the high aspect ratio region. Accordingly, the aspect ratio is preferably from 10 to 30, and more preferably from 15 to 25. The extinction ratio is defined as the energy ratio, expressed in the unit of decibel, of the transmitted light in the direction X and that in the direction Y when nonpolarized input light of the specified wavelength is used; when the energy ratio is 10, the extinction ratio is 10 dB. If the minor axis lengths of the metal particulates 5 increase, the insertion loss of the polarized light in the direction X to be transmitted will increase. In view of this point as well, the aspect ratio is preferably 10 or over, and more preferably 15 or over, and preferably, the minor axis length is short and the insertion loss is small. If the mean major axis length of the metal particulates 5 increases, the peak wavelength of absorption in the direction Y will increase and come closer to the wavelength range used by the optical communication (about 1.3μm). However, it is taken into consideration that the aspect ratio of the metal particulates has production-related limitations and the increase in the minor axis length invites insertion loss, the length of the major axis itself has a limit.

[0023] The conditions for the metal particulates are preferably that the aspect ratio is from 10 to 30, that the average of the major axis length is from 10 to 300 nm, and that the average of the minor axis length is from 1 to 10 nm; more preferably that the aspect ratio is from 10 to 30, that the average of the major axis length is from 30 to 200 nm, and that the average of the minor axis length is from 2 to 10 nm; and most preferably that the aspect ratio is from 15 to 25, that the average of the major axis length is from 40 to 200 nm, and that the average of the minor axis length is from 2 to 10 nm.

[0024] In the case of Fig. 1, the polarized light component in the direction Y of the incident light 6 entering in the direction Z is absorbed by the resonance with the free electrons of the metal particulates 5, and the polarized light component in the direction X has a high transmissivity and becomes the polarized outgoing light 7. There is a difference in the peak wavelength of absorption between the direction X and the direction Y; in the direction Y the peak of absorption is at a longer wavelength than that of the direction X. When not particularly specified, the above-mentioned extinction ratio is determined by the wavelength at which the peak of absorption occurs in the direction Y.

Experiment

[0025] Pyrex #7740 (Pyrex #7740 is a trade name of Corning Glass Industry Inc.) was used for the glass substrate 2. The composition of the glass substrate 2 is SiO_2 80.8 %, Al_2O_3 2.3 %, B_2O_3 12.5 %, Na_2O 4.0 % and the balance being minor components such as Fe_2O_3 and K_2O; the composition is given by weight percent. Its softening point is 820 °C. The size of the substrate 2 is 76 mm long, 10 mm wide and 1 mm thick.

[0026] To achieve 10 vol. % of the metal content in the polarizing layer 3, Pyrex glass (the same Pyrex #7740 as the substrate 2) and Au were used as targets, and Au as metal particulates and Pyrex as the dielectric were simultaneously sputtered on the substrate 2 by dual magnetron sputtering. The sputtering conditions included that the RF power was 20 W, the sputtering gas was Ar and the pressure was 2.0×10^{-3} Torr, and the flow rate of Ar was 10 cc/m.

[0027] The polarizing layer was formed as a single film, and its film thickness was 0.5 μm before the drawing. The substrate 2 after the film formation was heat-treated for one hour at 700 °C in the atmosphere to make the metal coagulate into metal particulates 5. The resulted metal particulates before the drawing had a mean grain size (diameter) of about 120 nm, and the grain size distribution was from 100 to 150 nm.

[0028] The spectral transmission under this condition

is shown in Fig. 2. The axis of ordinates indicates the extinction ratio showing the ratio of the incident light 6 to the outgoing light 7 in the unit of dB. The axis of abscissas shows the wavelength used. There is a peak of absorption of about 20 dB near 0.5μm. Next, the substrate 2 was drawn by applying forces of 45 kg/mm^2 on both the ends of the substrate in opposite directions. The drawing conditions are preferably that the force is from 10 to 100 kg/mm^2, that the temperature at the time of drawing is from 500 to 800°C, and more preferably that the temperature is from 650 to 700 °C. The preferable range of amount of drawing is from 40 to 300 mm in drawing length for the substrate 2 of 76 mm long. This means that the drawing rate is from 50 to 400 %. In the embodiment, forces of 45 kg/mm^2 were applied on both the ends of the substrate 2 at 675°C in the atmosphere to draw the substrate 2 by 50 mm. As a result, the film thickness of the polarizing layer 3 became about 0.3 μm. In the polarizer thus obtained, the aspect ratio of the metal particulates 5 was 20 in average, and the major axis length thereof was about 80 nm±10 nm, and the minor axis length thereof was about 4 nm ± 2 nm. The extinction characteristics of the polarizer obtained are shown in Fig. 3. The axis of abscissa shows the wavelength, and the axis of ordinates shows the extinction ratio. 8 denotes the extinction ratio of the light polarized in the direction X of Fig. 1, and 9 denotes the extinction ratio of the light polarized in the direction Y of Fig. 1. As is clear from Fig. 3, at the wavelength of 0.55μm, an extinction ratio of 20 dB was achieved.

[0029] Next, the polarizers 1 thus obtained were arranged to sandwich a Farady rotator, magnets were coaxially arranged around the Farady rotator, and they were put into a Ni-Fe holder to produce an optical isolator. Sealing glass was used to hermetically seal the polarizers 1, 1 in the holder. The sealing temperature was 500°C, and because the coefficient of thermal expansion of the substrate 2 and that of the holder were close to each other, hermetic sealing was made successfully.

Claims

1. A production method of an optical polarizer having a transparent dielectric substrate and at least a polarizing layer comprising a glass component and metal particulates provided on a principal face of the substrate, the method comprising:

   forming the polarizing layer; heating the substrate and the polarizing layer for making metal particulates in the polarizing layer coagulate; and
   drawing the substrate and the polarizing layer so that the metal particulates in the polarizing layer have a shape anisotropy;

   characterised in that the polarizing layer is produced by simultaneous sputtering of the glass component and the metal particulates onto the substrate so that the polarizing layer is produced as one single thin film where the metal particulates are homogeneously dispersed in said glass component.

2. A production method according to claim 1 **characterised by** both said dielectric substrate and said glass component being borosilicate glass.

3. A production method according to claim 1 **characterised by** said metal particulates being at least one member of a group consisting of Au, Pt, Rh, Ir, Cu, Fe, Ni, and Cr.

4. A production method according to claim 1 **characterised by** said metal particulates having an aspect ratio ranging from 10 to 30 in average.

5. A production method according to claim 4 **characterised by** said metal particulates having a mean major axis length of 10 to 300 nm and a mean minor axis length of 1 to 10 nm.

6. A production method according to claim 1 **characterised in that** the metal particulate content in the polarizing film is 5-15%.

7. A production method according to claim 1 **characterised by** said metal particulates being at least one member material of a group consisting of Au, Cu and their alloys.

8. A production method according to claim 1 **characterised in that** said one single film has a thickness of 1 μm or less.

Patentansprüche

1. Verfahren zur Herstellung eines optischen Polarisators mit einem durchsichtigen dielektrischen Substrat und mindestens einer Polarisierungsschicht, welche eine Glaskomponente und Metallteilchen umfasst, die auf einer Hauptfläche des Substrates vorgesehen sind, wobei das Verfahren folgende Schritte aufweist:

   Bilden der Polarisierungsschicht; Erwärmen des Substrats und der Polarisierungsschicht, um Metallteilchen in der Polarisierungsschicht zur Koagulierung zu veranlassen, und
   Ziehen des Substrats und der Polarisierungsschicht in der Weise, dass die Metallteilchen in der Polarisierungsschicht eine Form-Anisotropie aufweisen,

dadurch gekennzeichn t, dass die Polarisierungsschicht durch gleichzeitiges Sputtern der Glaskomponente und der Metallteilchen auf das Substrat in der Weise hergestellt wird, dass die Polarisierungsschicht als ein einziger dünner Film gebildet wird, in welchem die Metallteilchen in der Glaskomponente gleichmäßig verteilt sind.

2. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass sowohl das dielektrische Substrat als auch die Glaskomponente aus Borsilikatglas bestehen.

3. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die Metallteilchen mindestens ein Element aus der Gruppe sind, die aus Au, Pt, Rh, Ir, Cu, Fe, Ni und Cr besteht.

4. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die Metallteilchen ein Seitenverhältnis aufweisen, das im Mittel im Bereich von 10 bis 30 liegt.

5. Verfahren nach Anspruch 4,
dadurch gekennzeichnet, dass die Metallteilchen eine mittlere Länge der größeren Achse im Bereich von 10 bis 300 nm und im Mittel eine Länge der kleineren Achse im Bereich zwischen 1 und 10 nm aufweisen.

6. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass der Gehalt an Metallteilchen in dem Polarisierungsfilm 5 bis 15 % beträgt.

7. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die Metallteilchen mindestens ein Materialelement aus einer Gruppe darstellen, die aus Au, Cu und deren Legierungen besteht.

8. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass der eine einzige Film eine Stärke von 1 µm oder weniger aufweist.

Revendications

1. Procédé de fabrication d'un polariseur optique ayant un substrat diélectrique transparent et au moins une couche polarisante comprenant un composant de verre et des particules métalliques prévue sur une face principale du substrat, le procédé consistant à :

   former la couche polarisante ; chauffer le substrat et la couche polarisante pour amener des particules métalliques dans la couche polarisante à coaguler ; et
   étirer le substrat et la couche polarisante de sorte que les particules métalliques dans la couche polarisante aient une anisotropie de forme ;

   caractérisé en ce que la couche polarisante est fabriquée par une pulvérisation simultanée du composant de verre et des particules de métal sur le substrat, de sorte que la couche polarisante est fabriquée comme une couche mince unique où les particules métalliques sont disposées de manière homogène dans ledit composant de verre.

2. Procédé de fabrication selon la revendication 1, **caractérisé en ce qu'**à la fois ledit substrat diélectrique et ledit composant de verre sont du verre au borosilicate.

3. Procédé de fabrication selon la revendication 1, **caractérisé en ce que** lesdites particules métalliques sont au moins un membre d'un groupe formé de Au, Pt, Rh, Ir, Cu, Fe, Ni et Cr.

4. Procédé de fabrication selon la revendication 1, **caractérisé en ce que** lesdites particules métalliques ont un rapport de forme compris entre 10 et 30 en moyenne.

5. Procédé de fabrication selon la revendication 4, **caractérisé en ce que** lesdites particules métalliques ont une longueur moyenne de grand axe de 10 à 300 nm et une longueur moyenne de petit axe de 1 à 10 nm.

6. Procédé de fabrication selon la revendication 1, **caractérisé en ce que** la teneur des particules métalliques dans la couche polarisante est de 5 à 15 %.

7. Procédé de fabrication selon la revendication 1, **caractérisé en ce que** lesdites particules métalliques sont au moins un membre d'un groupe formé de Au, Cu, Fe et leurs alliages.

8. Procédé de fabrication selon la revendication 1, **caractérisé en ce que** ladite couche unique a une épaisseur de 1 µm ou moins.

FIG. 1

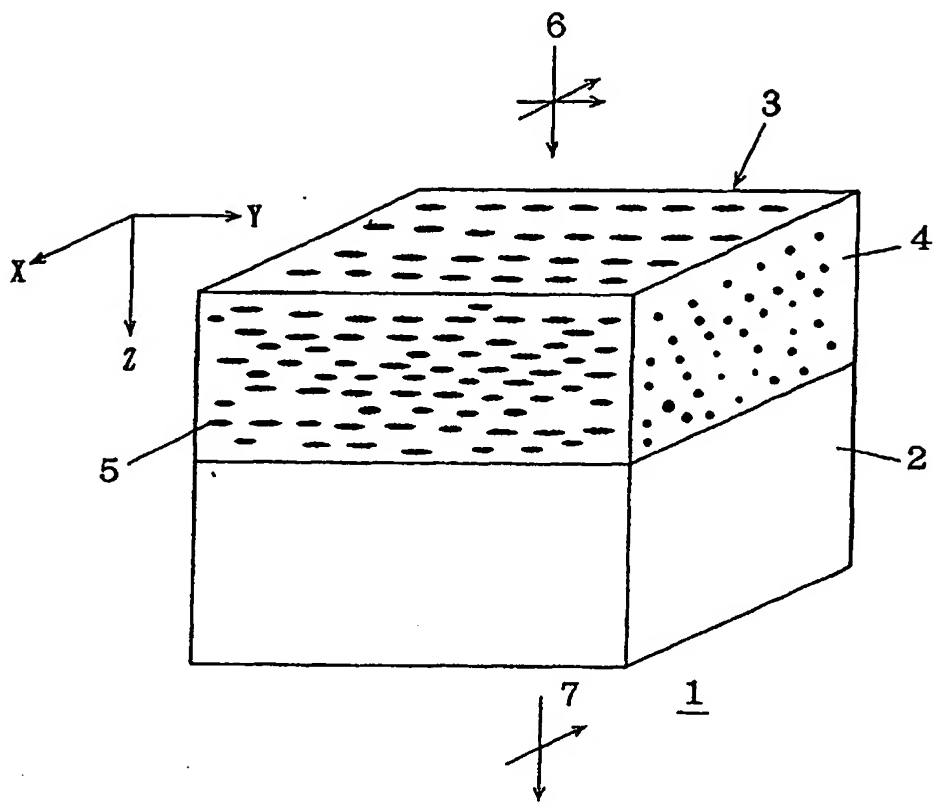

FIG. 2

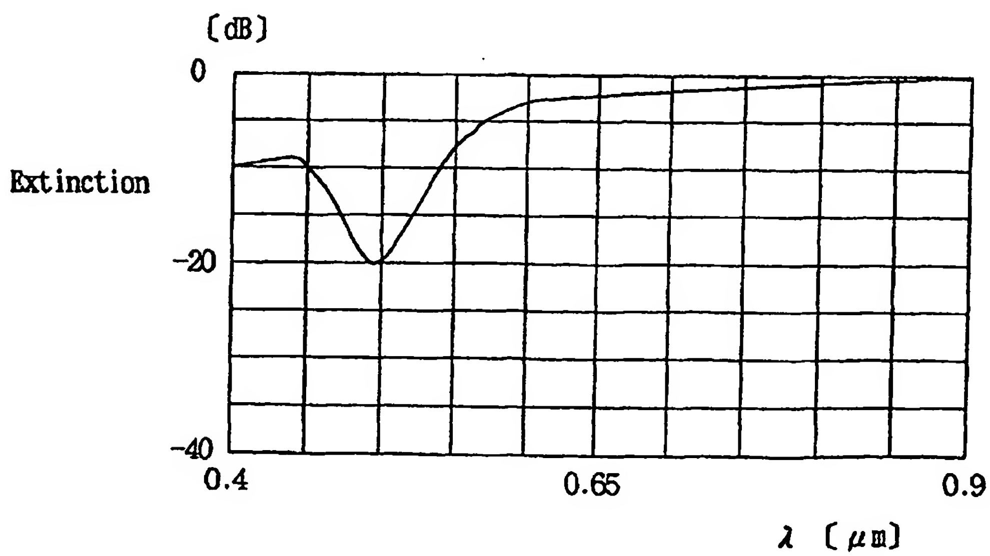

FIG. 3